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SUBSURFACE DEFECT DETECTION IN CERAMIC MATERIALS USING LOW COHERENCE OPTICAL SCATTER REFLECTOMETER

Mark Bashkansky, Michael D. Duncan, Manfred Kahn*, David Lewis III† and J. F. Reintjes‡

Laser Physics Branch, Code 5640
Naval Research Laboratory
4555 Overlook Ave. SW
Washington, DC. 20375
Tel: (202) 767-2512 Fax: (202) 404-7530

Abstract: We demonstrate the use of optical gating techniques for determining the size and location of subsurface defects in advanced ceramic materials. Various silicon nitride based ceramic materials are probed non-destructively using an optical gated reflectometer based on a low-coherence fiber interferometer. This device is capable of depth and lateral resolutions of 10 μm and 4 μm, respectively. Experimental results indicate that the size and position of small subsurface defects can be determined as deep as 500 μm below the surface.

Key Words: Ceramic; nondestructive evaluation; optical gating

Introduction: Advanced ceramic materials have great potential in industrial and military applications because of their superior thermomechanical and chemical properties. However, their widespread use is limited in part due to the presence of defects in both the bulk and on the surface of the ceramic. Defects are manifested as voids and cracks, or as inclusions which are caused by inherent powder defects or poorly distributed second-phase material. Such defects can be introduced at various stages of the manufacturing process. Potentially, these defects can reduce structural integrity of the ceramic components and lead to variability in their material properties, both of which may be unacceptable in critical applications. Because the potential market for ceramic components is so large, a considerable amount of effort has been put into developing techniques to detect flaws at all stages of the manufacturing process[1-6].

Defects that exist near the surface are particularly critical in many applications, since operational stresses in this region of the ceramic component can be greatest. Therefore, flaws in the vicinity of the surface can compromise the strength, degrade performance and shorten lifetime of a ceramic component. These flaws may be intrinsic to the bulk material, introduced in the final stages of fabrication (e.g. machining, grinding and polishing) or appear during the operation of the device.

Recently, several ultrasonically based non-destructive evaluation (NDE) techniques have been applied to the detection of surface and subsurface damage in ceramic materials[3-5]. In photoacoustic microscopy an amplitude-modulated laser beam is used to excite the acoustic modes in the sample[4]. A transducer, attached to the sample, is used to detect the frequencies and the amplitudes of the acoustic modes. Even though, in principle, this measurement can yield information about the flaws in the surface and near surface regions, this technique has had difficulty in detecting smaller flaws and adequately shielding the device from other sources of noise. In pulse-echo NDE, the acoustic modes are excited by a highly focused ultrasonic pulse[5]. By focusing the ultrasound to a spot below the surface some of the pulse energy goes into exciting a surface mode of the ceramic. To date, to our knowledge, no distinguishing signatures have been found in the return echo which can be conclusively associated with subsurface damage.

An alternative to acoustic techniques has recently been developed where the polarization properties of the reflected light are utilized for defect detection in ceramics[6]. As light propagates through the ceramic it is both absorbed and scattered. The scattering occurs at the grain boundaries and interfaces with the second-phase material, where the index of refraction changes discontinuously. To distinguish between surface and subsurface reflections, both of which have different light scattering characteristics, a NDE technique using a cross-polarization method was developed[6]. If the surface of the ceramic is optically polished then the light that reflects from the surface will, for the most part, retain the polarization of the incident beam. In this case, an analyzing polarizer placed before the detector can be oriented to block most of the reflected light from the surface. However, the light that is scattered by the irregular defects below the surface is most likely depolarized and, therefore, is partially transmitted through the polarizer. Depending on the orientation of the analyzing polarizer, the light is detected, for the most part, from either the surface or the subsurface region. An advantage of optical techniques over ultrasonic techniques is that no contact with a sample is required. However, the limitation of the cross-polarization method is that it cannot determine directly how far below the surface a defect is located, or accurately measure how large the defect is. This information is important because defects that are either far below the surface or smaller in size may result in a fewer failures than large defects or defects near the surface.

Recently, a variety of ultra-fast optical range-gating techniques have been developed for potential biomedical applications[7-10]. In general, these techniques utilize the fact that photons which scatter below the surface travel farther to reach the detector than photons that scatter from the surface. Therefore, when a short optical pulse scatters from a sample, the detected return will be spread out in time. There will be a near one-to-one correspondence between the time the light arrives at the detector and the depth from which it came. Thus, by temporally resolving the intensity of the scattered light it is possible to determine the depth location of defects in the sample. Furthermore, it is possible to utilize either very short light pulses or long pulses with very short coherence length. These techniques have demonstrated depth resolutions in air $\leq 10\ \mu m$.

In this work, we applied an optically-gated reflectometer that is based on a low coherence fiber interferometer[11,12] to the detection of both the location and the size of defects in the subsurface region of ceramic components. This device has advantages of simplicity and low cost.

Experimental Setup: The experimental set up for detecting subsurface defects in ceramics is shown schematically in Fig. 1. Optical radiation at 1.3 μm is produced by a light emitting diode (LED). The full-width-at-half-maximum (FWHM) of the spectral content of the radiation was measured to be ~ 40 nm at an output power of 130 μW. The FWHM of the correlation length of this LED in air (equivalent to the depth resolution) is a function of the spectral content of the radiation and for a Gaussian spectrum is given by[9] $\Delta L = \ln(2)\frac{2}{\pi}\frac{\lambda^2}{\Delta\lambda} \approx 20\mu m$.

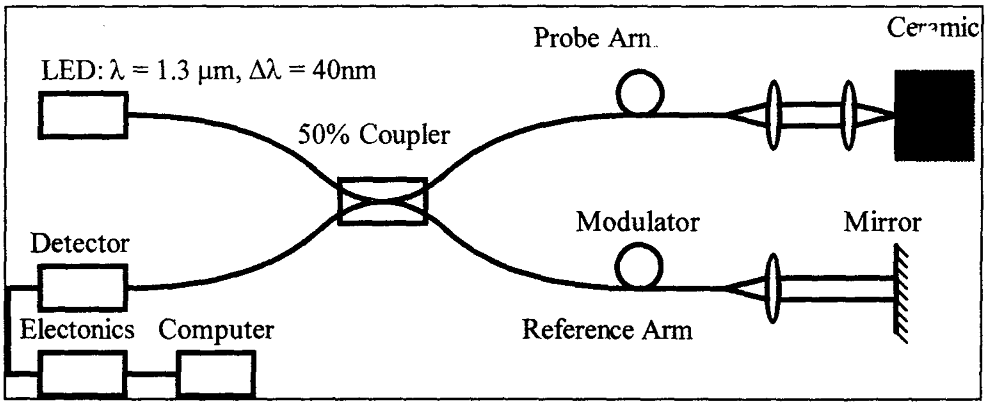

Figure 1

Experimental schematic of the low-coherence fiber interferometer system used to detect subsurface defects in ceramic material.

The LED radiation is coupled to a fiber and split into reference and probe beams in a 50 % fiber coupler. The phase of the reference beam is modulated by stretching the fiber with a piezoelectric transducer. The reference beam is retro-reflected from the mirror back into the fiber and toward the 50 % splitter. The probe beam is focused on the ceramic sample. The probe radiation scattered from the ceramic retraces its path through the fiber and recombines with the back-propagating reference beam in the 50 % splitter. This combined radiation is detected by a fiber-coupled photodiode. The spatial extent of the probe beam's focal spot was measured to be ~7 μm (FWHM). This gives resolution roughly 1/5 of that allowed by the diffraction limit at 1.3 μm.

When the pathlength difference between phase-modulated reference and probe beams is within the coherence length of the radiation, the interference between them produces an alternating current (AC) component on the photodiode signal. The frequency of this AC component depends both on the frequency and the depth of the reference beam modulation. This AC component represents the range-gated signal and its magnitude is proportional to the amount of light reflected from the ceramic. The photodiode signal is AC coupled, and rectified such that a direct current (DC) signal is produced proportional to the root-mean-square (RMS) of the AC signal. The logarithm of this DC signal is stored in a computer after analog-to-digital conversion.

By adjusting the relative depth (Z) position of the ceramic, the light that is scattered from different depths of the ceramic will become correlated with the reference beam. Only the correlated component of the scattered probe beam contributes to the AC signal on the photodiode. Thus, by varying the X-Y-Z position of the ceramic at the focus of the probe beam, the sizes and locations of the defects in the ceramic material can be mapped in three dimensions.

RESULTS: The first ceramic material used in this study was a hot, isostatically pressed (HIPed) silicon nitride, designated NCX 5102, which was obtained from Saint-Gobain/Norton Industrial Ceramics Corp. This ceramic is primarily silicon nitride (Si_3N_4) with a small percentage of Y_2O_3, a second phase material.

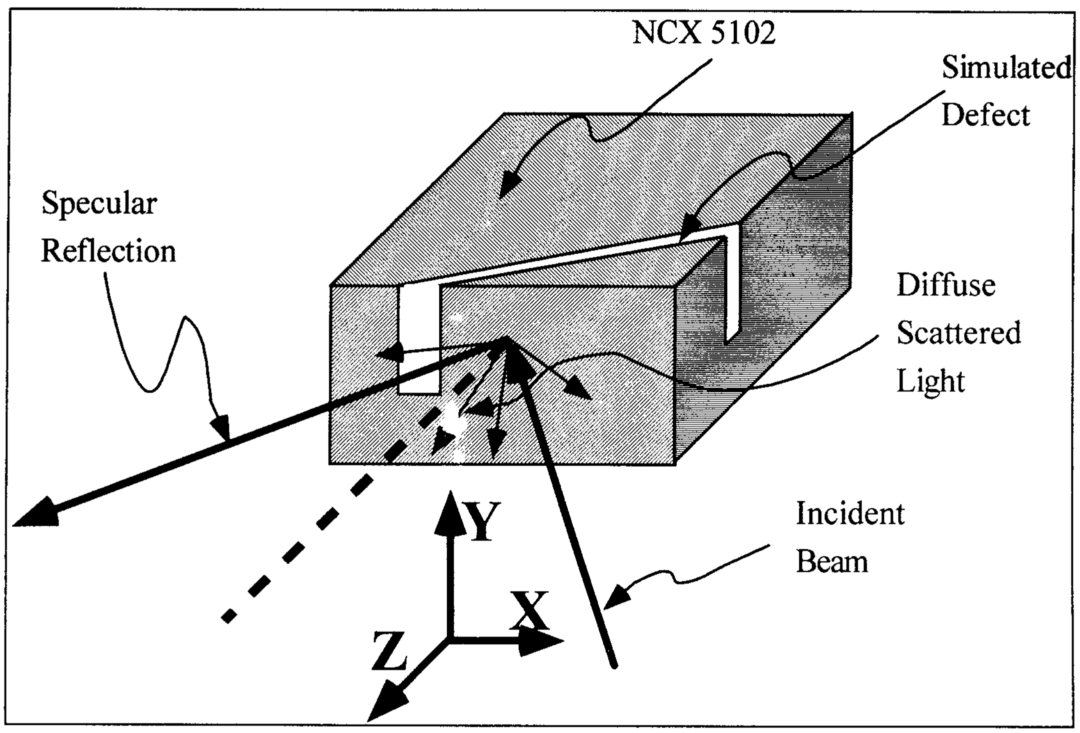

Figure 2

A schematic diagram shows the geometry used for subsurface defect detection in a ceramic material. The probe beam is incident at an angle with respect to the front surface of the ceramic so that the specular reflection is rejected by the imaging system. A saw cut was made in the ceramic at a slight angle to the front surface in order to simulate a void-like defect at varying depths.

NCX 5102 was developed for high temperature applications such as in components of gas turbine engines. The choice and concentration of second phase material has a significant effect on the optical properties of a ceramic[6].

The dimensions of the ceramic sample are 3x4x10 mm. The 3x4 mm surface is positioned at an angle to the incident signal beam so that the specular reflection from the surface is not collected by the imaging system, as shown in Fig. 2. A subsurface defect is simulated in the sample by cutting a grove approximately 200μm wide and 2 mm deep. The saw cut is at a small angle to the surface so that as the ceramic is translated horizontally in the X-direction the void, as seen by the probe beam, moves deeper into the ceramic.

Fig. 3 shows an X-Z image through the sample reconstructed from the individual range-gated scans in Z. As the probe beam propagates through the ceramic it is both scattered and absorbed.

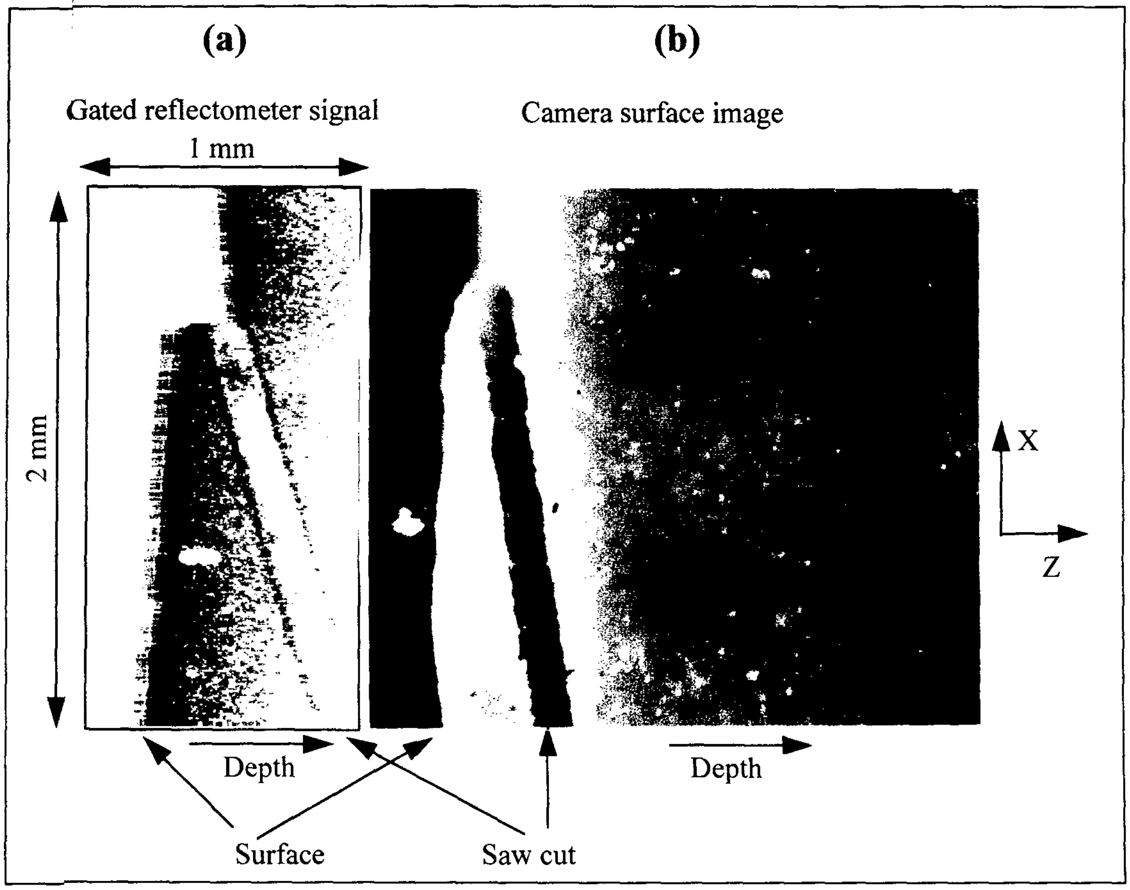

Figure 3

(a) An X-Z image through the middle of the NCX 5102 sample reconstructed from the individual depth scans in Z. (b) A camera image of the top surface of the ceramic, showing the saw cut.

The scattering results from grain-boundary reflections which are enhanced by the refractive index mismatch, Δn, of the second phase material. In the absence of voids or defects, the return from the scattered light decreases with depth into the ceramic. We see the reduction in signal level below the surface as expected. We also clearly see the increased scattering from the front and back surfaces of the saw cut. Current experimental conditions define the depth at which the void can be detected to about 500 μm below the surface.

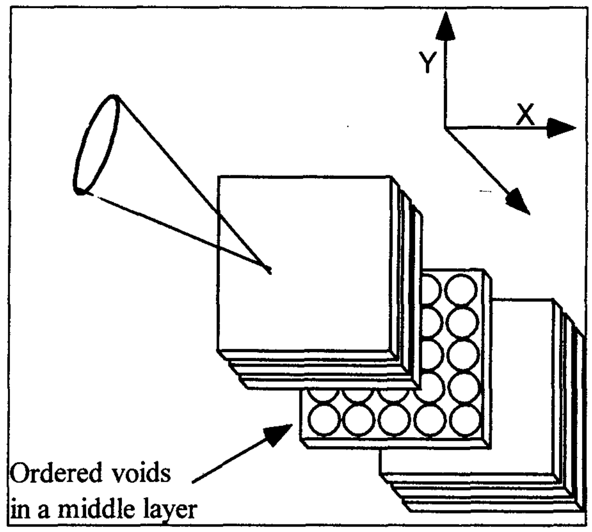

Figure 4

A schematic diagram showing internal structure of the ceramic composite material with ordered voids. Individual ceramic layers are 25 μm thick and the circularly shaped voids are present in some layers below the surface.

The second object we studied was a composite structure made from a ceramic kr`wn as PZT 5A. It is advantageous unc .r some circumstances to use multi-layered components rather than single piece devi^es. This structure, as shown in Fig. 4, was made at NRL from multiple ceramic layers and was provided with an ordered array of holes in some layers.

Fig. 5 (a) shows an X-Z image through the above structure reconstructed from the individual range-gated scans in Z. We can clearly see the individual layers below the surface. The dark regions in the fourth layer can be attributed to the voids. These regions are followed by increased signal in the fifth layer which we attribute to the void to ceramic interface. To see the voids below the surface directly, we performed a scan in the X-Y plane with the light focused on the fifth layer in Z. The results are shown in Fig. 5 (b). The circular voids are well defined in the range-gated image.

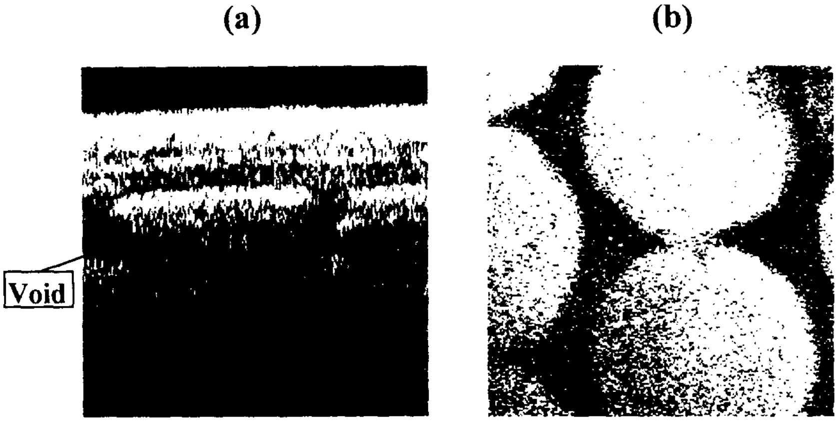

Figure 5

(a) An X-Z image through the ceramic composite structure, reconstructed from th individual depth scans in Z. The individual layers are clearly _ en. (b) An X-Y image of the fifth layer below the surface of the same structure.

SUMMARY: In conclusion, we have demonstrated the use of a range-gated reflectometer using scattered light for the detection of subsurface defects in ceramic materials. In particular, we have shown that subsurface voids as deep as 500 μm can be detected with a depth resolution of approximately 10 μm. The range-gating technique used in this work is based on a low-coherence fiber interferometer. An advantage of optical-gating techniques over non-gated light scattering techniques is that they not only detect the presence of a defect but also determine its depth below the surface. Future work will include characterizing the size and type of defects which can be detected using optical-gating techniques in various ceramic materials. We will explore the relation between fabrication and finishing and crack formation in various ceramic materials. In addition, we will extend the optical wavelength further into the IR, in order to increase the depth at which defects can be detected.

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